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IS 13605 (1992): Method for macroetch test for zinc and zinc alloys [MTD 22: Metallography and Heat Treatment]

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जस्ता और जस्ता मिश्रण के लिये स्थूल रसोत्कीणन की पद्धति

Indian Standard

METHOD FOR MACROETCH TEST FOR ZINC AND ZINC ALLOYS

UDC 669·55·620·183·255

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 1

Metallography and Heat Treatment Sectional Committee, MTD 22

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Metallography and Heat Treatment Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Macroetching is used to reveal the heterogeneity of metals and alloys. Metallographic specimens and chemical analysis will provide the necessary detailed information about specific localities but they cannot give data about variation from one place to another unless an inordinate large number of specimens are taken.

Macroetching, on the other hand, will provide information on variations in: (1) structure, such as grain size, flow lines, columnar structure, dendrites, etc, and (2) variations in chemical composition as evidenced by segregation. The information provided about variations in chemical composition is strictly qualitative but the location of extremes in segregation will be shown. Chemical analysis or other means of determining the chemical composition would have to be performed to determine the extent of variation. Macroetching will also [redacted] show presence of discontinuities and voids, such as seams, laps, porosity, flakes, bursts, extrusion rupture, cracks, etc.

In the preparation of this standard assistance has been derived from ASTM E340 Standard Test Methods for Macroetching Metals and Alloys, American Society for Testing and Materials.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHOD FOR MACROETCH TEST FOR ZINC AND ZINC ALLOYS

1 SCOPE

Macroetching is the etching of specimen for examination with unaided eye at low magnifications and is used to reveal the macrostructure of zinc and zinc alloys products, such as bars, billets, forgings and castings. The present procedure is intended to be followed for classifying, zinc and zinc alloys, characteristic structure and incidence of certain processing defects and is applicable to zinc and zinc alloy products.

2 SIGNIFICANCE

Macroetching is a simple test which provides information about the heterogeneity of the sample. It will show: (a) variation in structure such as grain size, flowlines columnar structure dendrite etc; and (b) presence of macro segregation.

3 SAMPLING

3.1 When using macroetching as an inspection procedure, sampling should be done in an early stage of manufacturing so that if the material proves defective, the minimum amount of unnecessary work is done. However, the sample should not be taken so early that further working can introduce serious defects. The sample is usually taken after ingot breakdown and after most chances of flaking and bursts have passed. Billets or blooms going into small sizes are sampled after initial breakdown. Materials going into forging billets or other products are sampled at near finish size, sampling may be done systematically or on a random basis.

3.1.1 Some common methods of sampling are listed as follows.

3.1.1.1 *Billets, blooms and hot-rolled products*

Discs are usually cut from these products near the end, but fish tails should be avoided. Discs from large blooms can be cut into smaller pieces for ease in handling.

3.1.2 *Forgings and Extrusion*

Discs, cut transverse to the long dimension, are used for examining bursts, voids, etc. Forging should be cut parallel to flash lines to show flow lines. Macroetching of an unprepared specimen shows surface defects, such as shuts, flats, seams, etc.

3.1.3 *Sheets and Plates*

While looking for defects in sheets, as large a sample as possible should be taken. When seeking information on lamination, a transverse section is used.

3.1.4 *Castings*

Specimen should be cut from different regions depending upon the features sought.

3.2 When macroetching is used to solve a problem, then the problem itself largely dictates the sample location on the workpiece and the stage of manufacture; for example, when looking for pipe, the sample should represent the top of the ingot, or when looking for burst, the sample should be taken as soon after hot working as possible.

3.3 Sample may be cold cut by any convenient means. Saw and abrasive cut off wheels and EDS cutting are particularly effective. The surface to be examined should be properly prepared before macroetching.

4 SPECIMEN PREPARATION

4.1 Sample preparation need not be elaborate. Any method of presenting a smooth surface with a minimum amount of cold work will be satisfactory. Discs may be faced on a lathe or a shaper. The usual procedure is to take a roughing cut, then a finish cut. This will generate a smooth surface and remove cold work layer from prior operations. Grinding may also be employed using a free cutting wheel and light finishing cut. When fine details are required, the specimen should be finished through the series of metallographic paper.

4.2 Sample preparation need not be elaborate. Any method of presenting a smooth surface with a minimum amount of cold work will be satisfactory. Discs may be faced on a lathe or shaper. The usual procedure is to take a roughing cut, then a finish cut. This will generate a smooth surface and remove cold work from prior operations. Grinding may also be employed using a fine wheel and light finished cut. When fine detail is required, the specimen should be finished with metallographic paper.

4.3 After surface preparation, the sample should be cleaned carefully with suitable solvents. Any grease, oil or other residue left on the surface will produce uneven attack during macroetching. Therefore, care should be taken not to touch the sample surface or contaminate it in any other way after it has been cleaned.

5 SOLUTIONS

5.1 The most common solutions for macroetching zinc and zinc alloys are given in Table 1.

Table 1 Macroetchants for Zinc and Zinc Alloys

(*Clauses* 5.1 *and* 6)

Alloy	Solution	Composition	Procedure	Comments
Cu-free zinc alloys	HCl (conc) H_2O	50 ml 50 ml	Immerse in solution for about 15 sec. Remove smut by wiping under running water. Repeat until desired contrast is obtained, then dry.	Grain structure
Zn alloy containing Cu	Cr_2O_3 Na_2SO_4 or $Na_2SO_4.10H_2O$ H_2O	200 g 1·5 g 3·4 g 100 ml	Immerse in solution until good contrast is obtained. Rinse in running water and dry. Repeat if necessary.	Grain structure

5.2 Caution must be observed in mixing the acids as many of the acids may cause chemical burns. The acid should be added slowly to water. Mixing and etching should be done in a fume hood, as many of the solutions are aggressive and may give irritating fumes.

6 PROCEDURE

The specimen shall be etched to reveal structure by immersion or by swabbing or by both means. The time of etching will vary depending upon the composition, size and structural condition. Generally speaking 30 s to 10 min will be Sufficient. Etching should be stopped when the preferred structural details have been revealed. Over-etching can lead to misinterpretation and hence light etching is better. Table 1 gives the time for various etchants, but should be used as a guide only. The actual time to develop a structure properly may be quite different from that suggested. After etching specimen should be washed in running water and blown dry with compressed air.

7 EXAMINATION OF SPECIMENS

7.1 Any special condition like segregation, banding, voids, etc, that can be observed should be reported. A low magnification (× 10) stereo microscope can be used for observation, if necessary. Normally it is essential to see the surface from various angles to observe flow defects and banding. The macrograin size for forged billets can be compared with specified macrostructures and level of grain size reported.

7.2 The acceptance and rejection criterion for these macrostructures has to be worked out according to the type of alloy and application. Therefore, the basis of macroetching inspection will be a matter of agreement between the manufacturer and the purchaser, if this technique is being used as a quality test.

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